Environmental Laboratory
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Information Update

June 19, 1996
Update #29

1. **Adding a method to a laboratory's current license.**

If a laboratory wants to add a method to their current license they need to do the following:

a. Request that Office of Environmental Laboratory Licensure add the promulgated method to their license.
b. Provide the Office of Environmental Laboratory Licensure with the following information:

   i. Current MDL study accompanied by hard copies of all the raw data.
   ii. The Initial Demonstration of Capability, including hard copies of all the raw data.
   iii. A copy of the laboratory's Standard Operating Procedure.
   iv. A copy of the results from the most recent appropriate WP or WS study that was performed by the requested method. If an appropriate WS or WP has not been performed, then contact the Office of Environmental Laboratory Licensure to arrange for a third party P/E sample.

c. Laboratory Licensure will assess a fee for the new method. The laboratory will need to pay this fee before their license can be changed to include the new method.

We recommend that all of the above information be sent to the attention of the individual from Arizona's Office of Environmental Laboratory Licensure who performed the last on site audit of the laboratory.

2. The methods 525.2 and 508.1 use the technique of solid phase extraction to extract the analytes from the matrix. These methods use a vacuum to filter the sample through the solid phase disk. It is however acceptable to use positive pressure to filter the sample. The lab needs to make certain that the sample is not being driven through the filter too fast. Being able to use positive pressure enables the laboratories to take advantage of the automated systems which are becoming available.
3. For drinking water, wastewater, and solid waste water samples a laboratory can use continuous liquid-liquid extraction as a replacement for separatory funnels.
4. As per Jean Munch, Research Chemist, USEPA/NERL, Cincinnati:

When running method 525.2 for phthalates for compliance purposes the lab must run a trip blank if any of the samples are found positive for phthalates. This is necessary to show that the samples were not contaminated with phthalates from the bottle caps, the HCL used for preservation or the latex gloves worn during sampling. Diethyl hexyl phthalates are of the main concern. If the samples show the presence of phthalates and there was no trip blank with the set of samples then, subsequent resamples from this site must be accompanied by a trip blank.

If the samples are not to be analyzed for phthalates then the lab does not need to run a trip blank.

The following information was provided by the staff of Environmental Laboratory Licensure Program:

a. The EPA Office of Solid Waste was consulted on the interpretation of Method 8270B sections 7.3.4, 7.3.5 and 7.6.2. According to the EPA, in order to use an average response factor, *all compounds* must have an %RSD of 15% for the initial calibration curve. If the %RSD is greater than 15%, then a first or higher order regression curve must be generated using the individual response factors. This curve must be used for quantitation. If the %RSD is less than 15% then either the average response factor or a first or higher order regression curve may be used. If the average RF %RSD of CCCs of the calibration curve are greater than 30%, neither the average RF nor a regression curve can be used; the calibration standards must be reinjected. This interpretation of the method will now be enforced by Laboratory Licensure.
b. As per clarification from EPA, the surrogate control limits in Table 8 of Method 8270B are to be used as guidelines and the laboratory may develop their own limits as long as they are reasonable and meaningful. For example, for Phenol-d_6 the surrogate spike recovery limits in water samples are 10-94 percent. If the laboratory has established their own range with a high limit of 100% , then the lab does not have to flag the data due to having exceeded the upper limit of 94% in Table 8.
c. It has been confirmed that section 7.6.8 of Method 8000A is not applicable to the mass spectrometry methods. The daily Calibration Check Verification (CCV) should be evaluated against criteria in the specific method. Because of the number of surrogates and internal standards required in these methods, an ending CCV is not required.
d. The Office of Solid Waste was also consulted about the corrective action to be taken when the ending CCV for GC and HPLC methods exceeds 15% difference. Method 8000A, section 7.6.8 does not address corrective action. It is the intent of the method that all samples be bracketed by CCVs to ensure that the instrument performed properly throughout the analysis sequence. Therefore if any CCV, continuing or ending, fails to be in control, all samples analyzed *before and after* the failed CCV should be reanalyzed. ADHS will allow the following exception to this: If the lab can demonstrate CCV specific failure (i.e. dry purge, carryover from previous sample) *and* the surrogates for the samples in the batch are in control, the samples before the failed CCV are acceptable, but the samples analyzed after the failed CCV must be repeated. Under no circumstances should data be accepted for samples that are analyzed without the CCV at the beginning of the analytical sequence being in control.

6. The Program of Environmental Laboratory Licensure is currently evaluating its current software needs. Please complete the attached survey and return to,

Attn: Roseann Pasqualone
State Laboratory Services
3443 N. Central Avenue, Suite 810

Phoenix, Arizona 85012

7. Technical Resources and Training will be facilitating a round table discussion on SOC and GC/MS methods. Some of our surveyors from the Environmental Licensure section as well as staff from State Laboratory will be available to answer questions and clarify problem areas via this session. There is no set agenda, it is a question and answer session. This will be held on Wednesday, **July 12, 1996, from 1:30-3:30 pm, at 3443 North Central, in the 9th floor conference room.** We request that laboratories fax questions to us prior to this date, in case we need to contact the EPA for further clarification. The areas to be covered are the approved methods, quality control, trouble shooting and maintenance of instrumentation or anything else about which you have questions. Please fax your questions to Prabha Acharya at 255-1070. This forum is NOT limited to questions sent before hand. Our training room can hold up to 35 people, so please RSVP with either Christy Finan or Amy Welch at 255-3454, if you are planning on attending.

Please note that we will not be validating any parking. Paid parking is available adjacent to the building on the street level.

8. Technical Resources and Training is considering hosting a one day workshop, where presenters from J & W Scientific would give the following four seminars.

    a. Care and Maintenance: How to Maximize Capillary Column Life.

    Capillary columns don't last forever; however, a number of techniques and practices will prolong their life. The cause, prevention and possible repair will be addressed for several different possible problems.

    b. Successful Pesticide Analysis: What You Should Know to Reduce Downtime.

    Topics covered in this presentation include injector setup and maintenance, the use of guard columns, press-fit unions, ECD maintenance and dual-column packages designed and tested specifically for routine organochlorine pesticide analysis.

    c. Optimizing Parameters Affecting Analysis of Volatile Organic Compounds by Purge and Trap.

    Topics include sample sparging, analyte trapping (including how to choose the right trap) and choosing a GC column that produces the best separation in the shortest run time possible. Also, there will be an opportunity to discuss maintenance for the P/T system, including detectors (PID, FID, ELCD, and MSD), and what can be done to prevent problems from occurring.

    d. Troubleshooting: When is it the (GC) Column's Fault?

When a GC problem occurs, it is often difficult to determine the actual cause. We all know that it is easy to be misled and reach the wrong conclusion. Systematic strategies for troubleshooting specific chromatographic problems will be presented, along with quick and easy tests to determine whether the column is the problem source. Many column problems are not caused by irreparably damaged column but by

some other factor interfering with the chromatography. These areas also will be addressed.

The workshop would be held sometime in early to mid August . We are estimating the cost to be $20.00 per person. Lunch and parking validation would be provided as part of the registration. In order to determine the feasibility of and the proper location for this workshop, we need to know how many people would be interested in attending. Please respond by fax to (602) 255-1070 if you might be interested in attending this workshop.

9. If you have any questions regarding the Updates or if you have any technical questions that need clarification, please call Prabha Acharya, Program Manager, Technical Resources and Training, at the above numbers.